

## Influence of light intensity on contraction stress of flowable resins

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**Abstract:** The purpose of this study was to evaluate the influence of power density on contraction stress of resin composite restorative materials during photopolymerization. Six flowable resin composites, and a hybrid resin composite for comparison, were used. The composites were polymerized with the power density adjusted to either 100 or 600 mW/cm<sup>2</sup>. Stress development was determined with a custom-made tensiometer. The adhesive was placed in a thin layer on a steel rod and resin paste was packed into the mold. The contraction force (N) generated during polymerization was continuously recorded and the maximum contraction stress (MPa) was calculated. Data were analyzed statistically. When the power density was adjusted to 100 mW/cm<sup>2</sup>, the average contraction stress ranged from 0.30 to 0.50 MPa for the flowable composites, compared with 0.35 MPa for the hybrid composite. When the power density was adjusted to 600 mW/cm<sup>2</sup>, the average contraction stress ranged from 0.34 to 1.00 MPa for the flowable composites and 0.69 MPa for the hybrid composite comparison. For all materials tested except Estelite Flow Quick, contraction stress increased with higher power density. The present results indicate that contraction stress during polymerization is influenced by power density and resin composite type. (*J. Oral Sci.* 50, 37-43, 2008)

**Keywords:** flowable resin; polymerization shrinkage; contraction stress.

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### Introduction

One major drawback of dental composite restoration is the shrinkage that accompanies polymerization (1). During polymerization, the intermolecular spaces between the dimethacrylate monomers are reduced by the conversion of C=C bonds, leading to volumetric shrinkage (2,3). The volumetric shrinkage of these materials causes stress within restored teeth and can create marginal gaps that influence the bonding of the restoration (4,5). Therefore, it is important to determine the polymerization characteristics of resin composites before their clinical use.

Flowable resin composites with easy handling properties have been developed, and are applied by means of a small-gauge dispenser (6-8). Manufacturers have developed them to meet the clinical requirements for improved functionality, especially with regard to their flow characteristics. Polymerization contraction stress is not only a function of a composite's volumetric shrinkage, but is also determined by its visco-elastic behavior. The visco-elastic behavior is usually described in terms of elastic modulus development and flow characteristics. In general, the higher the volumetric contraction, the higher the stress will be. As flowable resin composites show specific flow characteristics, the filler contents are relatively low compared with those of hybrid resin composites (9). Therefore, these materials might show different contraction behaviors during polymerization compared with those of hybrid-type resin composites.

The light irradiance provided by the curing unit may

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influence the polymerization contraction stress developed by resin composites (10). Activation with a high power density triggers a rapid polymerization process, leading to higher contraction stress in the composite (11). On the other hand, polymerization with lower power density may reduce leakage development at the tooth-restorative material interface (12). Although the power density of the curing unit may affect the contraction stress development of flowable resins, little is known about this aspect. The

purpose of the present study was to determine the influence of power density on the contraction stress of flowable resins. The null hypothesis to be tested was that the contraction stress of a flowable resin composite was not influenced by changes in the power density of a curing unit.

## Materials and Methods

### Resin composites and curing unit

The flowable resins used in this study are listed in Table 1.

A visible-light curing unit (Optilux 400; Demetron/Kerr, Danbury, CT, USA) was connected to a variable-voltage transformer (Type S-130-10; Yamabishi Electric Co., Tokyo, Japan) in order to adjust the power density to either 100 or 600 mW/cm<sup>2</sup>; these values were determined using a dental curing radiometer (Model 100; Demetron/Kerr).

### Contraction stress measurement

Stress-strain data for the light-activated resin composite during curing were obtained by means of a dynamic test method performed on a custom-made tensiometer connected to a universal testing machine (Autograph AG-I, Shimadzu Corp., Kyoto, Japan). The composite was bound to a brass rod with a specimen holder, and the whole of the setup was connected to the stationary part of the framework (Fig. 1).

The composite was inserted into a mold, creating a configuration factor (C-factor,  $C = d/2h$ , where  $d$  and  $h$  are the diameter and thickness of the disk samples, respectively)

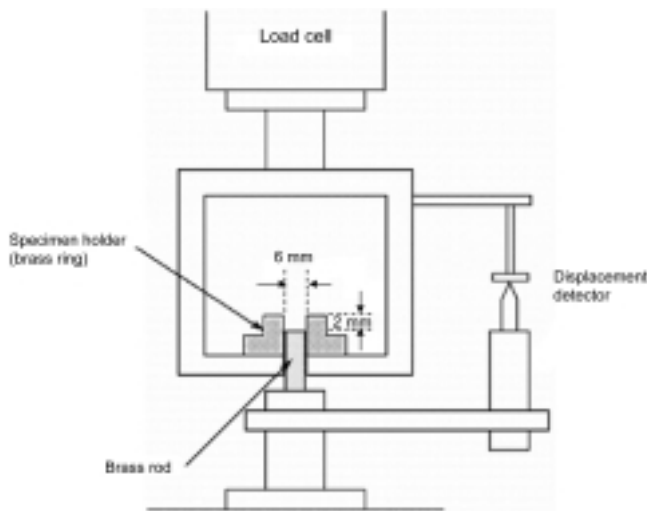


Fig. 1 Contraction stress measurement system used in this study.

Table 1 Materials tested

Composite	Lot No.	Filler type and contents	Manufacturer
Beautiful Flow F02	110405	S-PRG filler, MF glass filler 48.7 wt%	Shofu Inc.
Beautiful Flow F10	120404	S-PRG filler, MF glass filler 48.0 wt%	Shofu Inc.
Clearfil Flow FX	00004A	Barium glass, Lanthanoid fluoride 61.3 wt%	Kuraray Medical Inc.
Estelite Flow Quick	J213	Silica-zirconia, Silica titania 57.8 wt%	Tokuyama Dental Corp.
Filtek Flow	4ALJ	Silica-zirconia 64.9 wt %	3M ESPE
Unifil Lo Flo Plus	507211	Fluoroalumino silicate 42.5 wt %	GC Corp.
Lite-Fil II A	100328	Splintered alumino-silicate, 79.6 wt%	Shofu Inc

S-PRG filler: surface-reaction-type pre-reacted glass-ionomer filler

MF glass filler: Multifunctional glass filler with a modified surface layer

for the specimen of 1.5. Optimal bonding between the resin composite and the steel disk was achieved by coating the brass rod surface with adhesive (One-Up Bond F Plus, Tokuyama Dental Corp., Tokyo, Japan), and was light-cured for 10 s. The steel disk of the displacement detector was moved down until it reached the pre-adjusted specimen height. When the distance between the upper and lower rods was decreased by shrinkage, the resulting current was amplified by the strain gage transducer and drove the servomotor to maintain a constant distance between the rods, and the developing force was measured by the load cell. Five specimens of each resin composite were evaluated. All measurements were performed at a constant temperature ( $23 \pm 1$ )°C and relative humidity ( $50 \pm 5$ )%.

### Statistical analysis

The mean and standard deviation (SD) were calculated and tested for homogeneity of variance using Bartlett's test. As the variances were found to be homogeneous, the data were subjected to analysis of variance (ANOVA) followed by Tukey's HSD test and Student's *t*-test ( $\alpha = 0.05$ ) using the Sigma Stat software program (Ver. 2.03; SPSS Inc., Chicago, IL, USA).

### Scanning electron microscopy (SEM)

The polished surfaces of the resin composites were observed by SEM. Resin pastes were inserted into a Teflon mold (6 mm in diameter, 2 mm in height) and cured for 40 s. The cured specimens were then polished with an Ecomet 4/Automet 2 (Buehler Ltd., Lake Bluff, IL, USA) using SiC papers with grit sizes of 600, 1,200, and 4,000,

followed by final polishing with a soft cloth with a grit size of 1.0  $\mu\text{m}$  and diamond paste (Buehler Ltd.). The polished surfaces were subjected to argon-ion beam etching for 30 s under an ion beam (Elionix Ltd., Tokyo, Japan) with an accelerating voltage of 1.0 kV and an ion current density of 0.4 mA/cm<sup>2</sup> directed perpendicular to the polished surface. The surfaces were coated with a thin film of gold (Au) in a Quick Coater Type SC-701 vacuum evaporator (Sanyu Denshi Inc., Tokyo, Japan), and observing using a field-emission SEM (ERA 8800FE, Elionix Ltd.).

## Results

The average maximum contraction stress of each composite is shown in Table 2. ANOVA revealed significant differences among the materials. When the resin composites were irradiated with lower power density (100 mW/cm<sup>2</sup>), contraction stress ranged from 0.30 to 0.50, and a significantly lower contraction stress was recorded for Estelite Flow Quick. When the resin composites were irradiated with higher power density (600 mW/cm<sup>2</sup>), contraction stress ranged from 0.34 to 1.00, and a significantly lower contraction stress was recorded for Estelite Flow Quick. Student's *t* test showed that there was a significantly higher contraction stress for all resin composites, except for Estelite Flow Quick, when irradiated with higher power density.

Representative SEM micrographs (backscattered electron images) of the composites evaluated in this study are shown in Fig. 2. Splintered filler particles of different sizes were seen in the first resin composite group: Beautiful Flow F02, Beautiful Flow F10, Clearfil Flow FX, and

Table 2 Contraction stress and filler content of resin composite

Composite	Contraction stress (MPa)	
	100 mW/cm <sup>2</sup>	600 mW/cm <sup>2</sup>
Beautiful Flow F02	0.34 (0.14) <sup>a</sup>	0.58 (0.14) <sup>c</sup>
Beautiful Flow F10	0.48 (0.07) <sup>a</sup>	0.72 (0.16) <sup>c,d</sup>
Clearfil Flow FX	0.50 (0.07) <sup>a</sup>	1.00 (0.12) <sup>c</sup>
Estelite Flow Quick	0.30 (0.05) <sup>b</sup>	0.34 (0.05) <sup>f</sup>
Filtek Flow	0.47 (0.10) <sup>a</sup>	0.81 (0.14) <sup>de</sup>
Unifil Lo Flo Plus	0.34 (0.03) <sup>a</sup>	0.66 (0.07) <sup>cd</sup>
----- Lite-Fil II A	0.35 (0.09) <sup>a</sup>	0.69 (0.05) <sup>cd</sup> -----

The values in parenthesis are the SDs. N = 5.

Within the same power density, the same superscript letteres indicate no significantly difference ( $P > 0.05$ ).

Significant increases in contraction stress were detected when the resin composite was irradiated with higher power density except for Estelite Flow Quick ( $P < 0.05$ )

Filtek Flow. Most of these particles were somewhat large ( $5\ \mu\text{m}$ ), while the remainder were smaller than  $1\ \mu\text{m}$ . Prepolymerized filler particles were observed in the second resin composite group: UniFil Lo Flo Plus. Splintered particles ( $1\text{-}5\ \mu\text{m}$ ) in conjunction with spherical and splintered prepolymerized filler particles ( $5\text{-}10\ \mu\text{m}$ ) were observed in the second group. Small spherical filler particles of different sizes were observed in the third resin composite group: Estelite Flow Quick. Particle size was typically  $0.4\ \mu\text{m}$ , the smallest particles being smaller than  $0.1\ \mu\text{m}$ . In the Lite-Fil II A specimen, different types of filler particles were observed as spherical agglomerated particles.

### Discussion

For more than a decade, several techniques have been developed and utilized for measurement of shrinkage strain. The dilatometer, linometer and bonded-disk methods

have been widely employed (13-15). However, progress in developing methods of comparable utility for determination of shrinkage-stress has been slower. Part of the difficulty resides in the design of the specimen holder, which must become bonded in the process of measurement. Though the premise underlying each of these testing methods is similar, the results for similar materials may vary greatly due to differences in the testing configuration, such as the direction of light application and specimen constraint (16).

Theoretically, volumetric shrinkage and elastic modulus are highly dependent on the material's filler content, although in opposite ways (9). Composites with a relatively high filler content have a low resin matrix fraction (Table 1), which actually determines the volume reduction observed during the formation of a dense cross-linked polymeric network. Shrinkage stress occurs when the

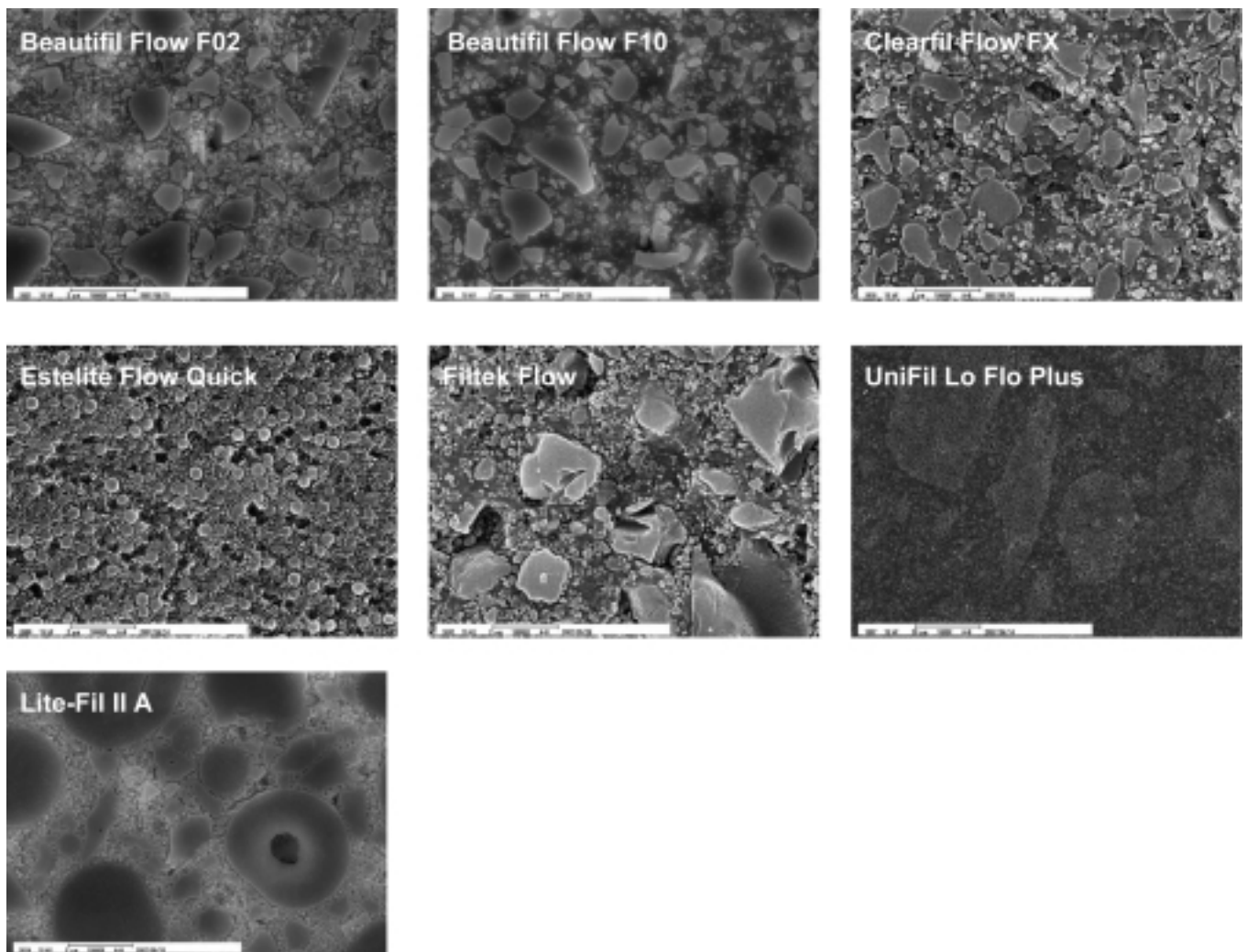


Fig. 2 SEM photographs of the ground surface of resin composites employed in this study. Original magnification was  $\times 10,000$  except for UniFil Lo Flo Plus, which was  $\times 1,000$ .



contraction is obstructed and the material is rigid enough to resist sufficient plastic flow to compensate for the original volume. Notwithstanding the strength of the materials involved, shrinkage will take place for the simple reason that it is physically unavoidable. Unless the surrounding structures to which the shrinking material is bonded supply enough elastic compliance, something will fracture in order to compensate for the reduced volume, and so create open spaces. The magnitude of the contraction stress is dependent not only on the compliance of the surrounding structures, but also on the nature of the shrinking material, notably its visco-elastic properties. At a given shrinkage value, the most rigid material is associated with the highest stress (17). The contraction stresses measured in this study might reflect this phenomenon.

The results of this study did not support the null hypothesis. Volumetric shrinkage of the flowable resins used in this study was influenced by the power density of the curing unit, except for Estelite Flow Quick. Contraction stress of a resin paste depends on factors such as filler loading, filler type, and filler size. Other factors that affect shrinkage behavior are the monomer and polymerization initiator systems because they determine the polymer structure of the material (18). Most light-cured materials use camphorquinone (CQ) as a photoinitiator (19). To initiate polymerization, an adequate intensity of visible light at wavelengths around 470 nm is required (20). Light of the appropriate wavelength is absorbed by the photoinitiator, which then reacts, in its excited state, with an amine-reducing agent to produce reactive free radicals. Working hand-in-hand, transmission of light through a light-cured material and the composition of the photoinitiator both influence the mechanical properties of the resin composite material. As for the power density of light passing through the material, it is controlled by the absorption and scattering of the latter's components (21).

The polymerization of resin composites is invariably accompanied by volumetric shrinkage of the cured material. Shrinkage is associated with the polymerization reaction in a complex way. Reduction in shrinkage strain can be attributed to reduced network connectivity, or to an increased propensity for flow of the material (22). Throughout the entire polymerization process, plastic deformation or flow of the resin composite may occur to a limited extent. Reduced power density may result in storage modulus development at a slow enough rate to allow for flow and dissipation of stress, while maintaining a sufficient bond to tooth structures (23). As the setting process proceeds, shrinkage and flow decrease gradually as storage modulus increases. With lower power density, flowable resins restrain the stress relief much more by not

allowing enough flow to reduce internal stress. Restriction of flow capacity by the configuration of the restoration also enhances contraction stress (24). In other words, marginal adaptation could be enhanced by optimal rheological effects. In particular, these beneficial effects of flow are being utilized when lower power density is employed with most flowable resin composites.

An irradiation unit with high power density is recommended based on mechanical properties (25). On the other hand, the possible negative influence of higher power density on stress development must also be considered. As polymerization shrinkage increases with higher power density, adverse effects on the marginal adaptation of resin fillings may occur. In the present study, higher power density resulted in an increase of contraction stress. Polymerization with a power density higher than those quoted by the manufacturers has been advocated to avoid areas of poor polymerization in the material. On the other hand, irradiation with a lower power density might retain some flow capacity within the polymerized specimen leading to lower mechanical properties. In light of these pros and cons of lower and higher power density of a curing unit, irradiation conditions appropriate to light-cured materials should be suggested and specified.

No significant difference in contraction stresses with differences in power density was found for Estelite Flow Quick, which employs a novel photopolymerization initiator system including a radical amplifier. It has been reported that the radical amplifier has higher polymerization activity than conventional polymerization initiators (26). The flowable resin composites used in this study were cured by a free radical polymerization reaction, and a photoinitiator such as CQ was employed. CQ required a coinitiator for an effective polymerization process to occur, and a tertiary amine photoreductant was employed (20). The tertiary amine interacted with an activated triplet state of CQ to form an intermediate excited complex, followed by production of reactive radicals for polymerization. To improve polymerization, an accelerator such as the radical amplifier was incorporated with the initiator system of Estelite Flow Quick. As a result, the irradiation time for this resin was reduced by 30% in comparison with the original light-cured resins (26).

The results of this laboratory study indicate that the contraction stresses of flowable resins are affected by power density and the resin composite type. With a higher power density, higher contraction stresses were recorded, except for a flowable resin that incorporated a radical amplifier. Further research is needed to investigate the behavior of flowable resin composites in clinical situations.

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